

#### ICF International / Laboratory Data Consultants

Environmental Services Assistance Team, Region 9 1337 South 46<sup>th</sup> Street, Building 201, Richmond, CA 94804-4698 Phone: (510) 412-2300; Fax: (510) 412-2304.

#### **MEMORANDUM**

TO:

Chris Lichens, Remedial Project Manager

Site Cleanup Section 4, SFD-7-4

THROUGH:

Rose Fong, ESAT Task Order Manager (TOM)

Quality Assurance (QA) Program, MTS-3

FROM:

Doug Lindelof, Data Review Task Manager

Region 9 Environmental Services Assistance Team (ESAT)

ESAT Contract No.: EP-W-06-041

Technical Direction Form No.: 00105041 Amendment 3

DATE:

March 16, 2007

SUBJECT:

Review of Analytical Data, Tier 3

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:

Omega Chem OU2

Site Account No.:

nt No.: 09 BC LA02

CERCLIS ID No.:

CAD042245001

Case No.:

Not Provided

SDG No.:

06-1647

Laboratory:

Applied Physics & Chemistry Laboratory (APCL)

Analysis:

1,2,3-Trichloropropane (1,2,3-TCP) and n-

Nitrosodimethylamine (NDMA)

Samples:

5 Water Samples (see Case Summary)

Collection Date:

March 6, 2006

Reviewer:

Nanny Estrada, ESAT/Laboratory Data Consultants

(LDC)

This report has been reviewed by the EPA TOM for the ESAT contract, whose signature appears above.

If there are any questions, please contact Rose Fong (QA Program/EPA) at (415) 972-3812.

Attachment

SAMPLING ISSUES: [X] Yes [] No

#### Data Validation Report – Tier 3

Case No.: Not Provided SDG No.: 06-1647

Site: Omega Chem OU2

Laboratory: APCL

Reviewer: Nanny Estrada, ESAT/LDC

Date: March 16, 2007

### I. CASE SUMMARY

Sample Information

Samples: OC2-MW7-W-0-155, OC2-MW7-W-4-156, OC2-

MW8C-W-0-157, OC2-MW8B-W-0-158, and OC2-

MW8A-W-0-159

Concentration and Matrix: Low Concentration Water

Analysis: 1,2,3-TCP (GC) and NDMA (GC/MS/MS CI)

SOW: EPA Methods 504.1 and 1625 Modified

Collection Date: March 6, 2006 Sample Receipt Date: March 6, 2006

Extraction Date: March 9 and 10, 2006 Analysis Date: March 10 and 14, 2006

Field QC

Field Blanks (FB): Not Provided

Trip Blanks (TB): OC2-MW7-W-4-156 (for 1,2,3-TCP only)

Equipment Blanks (EB): Not Provided Background Samples (BG): Not Provided Field Duplicates (D1): Not Provided

Laboratory QC

Method Blanks & Associated Samples:

06G1343MB01: (1,2,3-TCP) All samples 06G1337MB01: (NDMA) All samples

**Tables** 

1B: Data Qualifier Definitions for Organic Data Review

#### Sampling Issues

On the chain of custody (COC), the "relinquished" date of 2/6/06 is incorrect; the correct date is 3/6/06 (see attached COC).

#### **Additional Comments**

For the NDMA analysis, decafluorotriphenylphosphine (DFTPP) was not analyzed. Since NDMA is analyzed by the chemical ionization (CI) technique, no adverse effect is expected.

This report was prepared in accordance with the following documents:

- ESAT Region 9 Standard Operating Procedure 901, Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Volatile and Semivolatile Data Packages;
- ESAT Region 9 Standard Operating Procedure 902, Guidelines for Data Review of Contract Laboratory Program Analytical Services (CLPAS) Pesticide/PCB Data Packages;
- EPA Method 504.1, 1,2-Dibromoethane (EDB), 1,2-Dibromo-3-chloro-propane (DBCP), and 1,2,3-Trichloropropane (123TCP) in Water by Microextraction and Gas Chromatography, Revision 1.1, 1995;
- EPA Method 1625C, Semivolatile Organic Compounds by Isotope dilution GC/MS, June 1989; and
- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.

#### II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

Parameter	Acceptable	Comment
Holding Time/Preservation	Yes	
GC/MS and GC Performance	Yes	
Initial Calibration	Yes	
Continuing Calibration	Yes	
Laboratory Blanks	Yes	
Field Blanks	Yes	
Surrogate (Method 504.1)	Yes	
Labeled Compound (Method 1625)	No	$\mathbf{C}$
Matrix Spike/Matrix Spike Duplicates	N/A	
Laboratory Control Samples/Duplicates	Yes	
Internal Standard	Yes	
Compound Identification	Yes	
Compound Quantitation	No	A, B
System Performance	Yes	
Field Duplicate Sample Analysis	N/A	
	Holding Time/Preservation GC/MS and GC Performance Initial Calibration Continuing Calibration Laboratory Blanks Field Blanks Surrogate (Method 504.1) Labeled Compound (Method 1625) Matrix Spike/Matrix Spike Duplicates Laboratory Control Samples/Duplicates Internal Standard Compound Identification Compound Quantitation System Performance	Holding Time/Preservation GC/MS and GC Performance Initial Calibration Continuing Calibration Laboratory Blanks Field Blanks Field Blanks Surrogate (Method 504.1) Labeled Compound (Method 1625) No Matrix Spike/Matrix Spike Duplicates Internal Standard Compound Identification Compound Quantitation System Performance Yes  Yes  Yes  Yes  Yes  No No System Performance Yes

N/A = Not Applicable

#### III. VALIDITY AND COMMENTS

- A. The following detected result is qualified as estimated and should be flagged "J".
  - NDMA in sample OC2-MW8C-W-0-157 (below the practical quantitation limit)

- Results below the practical quantitation limits (PQLs) are considered to be qualitatively acceptable, but quantitatively unreliable, due to the uncertainty in analytical precision near the limit of detection.
- B. The laboratory reported the NDMA sample practical quantitation limit (PQL) as 0.002 ug/L and reported a NDMA detected result of 0.0009 ug/L for sample OC2-MW8C-W-0-157. However, the signal to noise (S/N) ratio is only 3 and the area is only 393 for the concentration of 0.0009 ug/L (see attached quantitation report, p. 2020 in data package). Furthermore, the area for low standard of the initial calibration is only 1074 (see attached quantitation report, p. 2029 in data package). In the reviewer's professional judgment, the sample PQL should be raised to 0.02 ug/L; non-detected sample results should be reported as 0.02U.
- C. The laboratory did not spike the samples and method blanks with a labeled compound (i.e., surrogate; see Method 1625C Sections 6.8, 10.2.1.3, and 10.2.3.2 and Figure 4). Consequently, the extraction efficiency (surrogate recovery) cannot be evaluated. The NDMA-d6 spiked by the laboratory was used as an internal standard.

#### TABLE 1B

## DATA QUALIFIER DEFINITIONS FOR ORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," October 1999.

- U The analyte was analyzed for but was not detected above the reported sample quantitation limit.
- L Indicates results which fall below the Contract Required Quantitation Limit. Results are estimated and are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in the analytical precision near the limit of detection.
- J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.
- UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.



# Applied P & Ch Laboratory

# Chain of Custody

13760 Magnolia Ave. Chino CA 91710 Tel: (909) 590-1828 Fax: (909) 590-1498

Please Print in pen

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	UCZ-MW7-W-4	-156 3/6/06/093	2 Water	HCL			$\mathcal{K}$					
	UC2-MW8C-W-	0-157 36106 1144	water	varies	6	X	XX					
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Target Compound #2 from 1647-4 f=0.00111.sms Sample ID: 1647-4 F=0.00111 Last Calibration: 2/21/2006 1:18 PM 3/14/2006 3:21 PM **Acquisition Date:** Calculation Date: 3/14/2006 2:52 PM c:\... \ndma005.mth Method: Vial: 3.0000 1.000000 Volume: Multiplier: Data File: c:\data\2006\06g1337\1647-4 f=0.00111.sms Compound Information Peak Name: N-Nitrosodimethylamine Identified Result Index: CAS Number: 62-75-9 Compound Number: 2 Identification **Parameter** Status Specification Actual Search Type Retention Time Retention Time 6.380 +/- 0.300 6.432 min. Pass Match Result N/A Integration and Quantitation Specification 75 **Parameter** Status Actual 4 6 1 Quan lons IS Peak Name NDMA-D6 Calibration Equation Linear, Force, None +0.7286x S/N Ratio **Peak Detection** Normal Slope Sensitivity 20 Peak Width 20.00 sec 4.3 sec **Baseline Type** Normal **Tangent Percent** 10% Area >=100 393 **Pass** Height 98 **Pass Amount** >= 0.000 ppb 0.810 ppb Sample Spectrum for Scan: 584 RT: 6.431 min. BP 75 (118=100%) 1647-4 f=0.00111.sms 6.431 min. Scan: 584 Chan: 1 lon: NA RIC: 233 100% 50% 92 54 89 Reference Spectrum for N-Nitrosodimethylamine Chan: 1 BP 75 (10000=100%) 50ppb.sms 6.409 min. Scan: 564 Ion: NA RIC: 18672 BC 100% 50% Sample Spectrum for Scan: 584 RT: 6.431 min. - Chan: 1 6.431 min. Scan: 584 Chan: 1 Ion: NA RIC: 233 Diff Reference Spectrum for N-Nitrosodimethylamine Normalized 50% 0% -50% -100% m/z Ion: 75 1 1647-4 f=0.00111.sms Counts RIC 1 1647-4 f=0.00111 sms 200 . 150 393 100

6.3

minutes

6<sup>1</sup>6

6.5

Print Date: 21 Feb 2006 13:42:46

# NDMA BY GC/MS/MS ION TRAP Operator: James Feng

Sample ID: **Acquisition Date:**  M005-002PPB

2/21/2006 10:20 AM

c:\... \ndma005.mth

Last Calibration:

Calculation Date:

Vial: Multiplier:

2/21/2006 1:18 PM

2/21/2006 1:18 PM

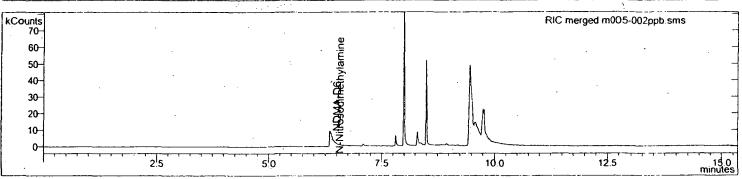
1.000000

Volume: Data File:

Method:

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## **Target Compounds**

# ≥	RT	Compound Name	Res Type	Quan lons	<u>Area</u>	<u>Amount</u>
1	6.363	NDMA-D6	Id.	81	50116	1.000 RRF
2	6.423	N-Nitrosodimethylamine	Id.	75 ·	1074	0.536 RRF

#### **Unidentified Peaks**

None